

Selected Grass Plants as Biomass Fuels and Raw Materials for Papermaking. Part I. Calorific Value and Chemical Composition

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Biomass yield was studied for tall wheatgrass, tall fescue, tall oatgrass, and *Miscanthus × giganteus* per hectare of cultivation, as well as their calorific value and cost of cultivating these biomasses. Chemical analyses were performed on these raw materials to determine their lignin, cellulose, extractives, and ash contents. The analytical results are compared to the chemical constituents found in birch and pine wood. It was found that the grasses examined in this study differed in biomass yield and cost to cultivate (1 ha plot). Tall wheatgrass, tall fescue, tall oatgrass, and *Miscanthus* leaves had lower levels of lignin compared with wood, but also lower amounts of cellulose. When determining the cellulose content of these biomasses, the amount of inorganic ash that is bound to them must be taken into account.

Keywords: Tall wheatgrass; Tall fescue; Tall oatgrass; *Miscanthus*; Calorific value; Chemical composition

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INTRODUCTION

The depletion of fossil fuel resources, decreasing forest area, deteriorating state of forest, and changes in the functions of forests towards an ecological direction are the main problems that humanity and the paper industry will face in the near future (Kozłowski *et al.* 1993; Ragauskas *et al.* 2006; Lucia 2008; Cherubini 2010). One solution to these problems is the wider use of biomass of non-wood crops to produce heat and electricity (Easterly and Burnham 1996; McGowin and Wiltsee 1996; Voivontas *et al.* 2001; McKendry 2002a; Khambalkar *et al.* 2008; Prochonow *et al.* 2009; Garcia *et al.* 2012; Protasio *et al.* 2013) and as fibre resources for papermaking (Guadalix *et al.* 1996; +Davis and Song 2006; Vargas *et al.* 2012; Ashley and Hodgson 2003; Jahan *et al.* 2014).

For non-wood biomass, one may include not only straw from energy crops and cereals, but also food industry products frequently found in municipal and industry waste (McKendry 2002b; Klass 2004; Mabee and Sadler 2010; Ho *et al.* 2014). Of these biomasses, cereal straw is by far the most accessible and widely used biomass for the production of heat in small boiler houses and pulps for papermaking (Leponiemi *et al.* 2010). Several reports have assessed the chemical composition, as well as pulp properties, of wheat straw (Pan and Leary 2000; Feng and Alen 2001; Guo *et al.* 2009; Hedjazi *et al.* 2009a; Saberikhah *et al.* 2011; Singh *et al.* 2011) and rice straw (Sun *et al.* 2000; Lam *et al.* 2001; Bhardwaj *et al.* 2005; Hedjazi *et al.* 2009b).

Some of the main problems associated with the use of cereal straw for energy generation and papermaking are the costs of transportation and storage, high silica content, and the unsuitability of existing pulp mill equipment for processing such raw materials (Delmas *et al.* 2003; Tutus and Eroglu 2004; Atik and Ates 2012). The usefulness of typical energy plants for burning (Shekhar Sharma *et al.* 2011; Christian *et al.* 2008; Godin *et al.* 2013a,b; Smith *et al.* 2015) and papermaking (Oggiano *et al.* 1997; Pahkala *et al.* 1997; Cappolletto *et al.* 2000; Ververis *et al.* 2004; Brosse *et al.* 2012; Monono *et al.* 2013) have also been studied. In Poland, this group of plants has also been evaluated as raw materials for energy purposes (Krasuska and Rosenquist 2012; Rozakis *et al.* 2013). Part I of this study presents the calorific values and chemical compositions of tall wheatgrass, tall fescue, tall oatgrass, and giant *Miscanthus*. These characteristics were examined to determine the suitability of these biomasses for combustion and papermaking.

EXPERIMENTAL

Types of Raw Materials and Soil Conditions of Cultivation

The following grass species were examined in this study: tall wheatgrass (TW; *Elytrigia elongata* (Host) Nevski); tall fescue (TF; *Festuca arundinacea* Schreb.); tall oatgrass (TO; *Arrhenatherum elatius* J. et C. Presl); and *Miscanthus* (*Miscanthus* × *giganteus* Gref et Deu.) – plant stalks (MS) and leaves (ML)). To produce new plantings of *Miscanthus*, three-year-old plants were divided while dormant (end of April, 2008) and the rhizome pieces were replanted. Planting density was one plant per m². Seeds of *Elytrigia elongata* were obtained from our own breeding of the ‘Bamar’ strain. Seed breeders kindly provided seeds of the tall fescue ‘Rahela’ and tall oatgrass ‘Wiwena’. These seeds were sown at distribution rates of 15, 11, and 17 kg·ha⁻¹, respectively. All species were sown in May 2008. Before sowing and planting, soil samples were taken at 0 to 30 cm in depth for the analysis of pH, N (NH₄), P, K, Ca, Mg, Cl, and soil organic carbon (SOC) contents. Soil properties at the beginning of the test are shown in Table 1. No additional treatments (fertilization, herbicides, or pesticides) were applied to the soil.

At the end of the growing season in 2010 (for tall wheatgrass, tall oatgrass, and tall fescue) or during February to March 2011 (for *Miscanthus*), the aboveground biomass was cut by hand and the biomass yields were measured. Biomass was further dried in an unheated greenhouse. Only *Miscanthus* plants were separated into leaves and stems. Typical wood chips used by the Polish papermaking industry, birch (*Betula pendula*), and pine (*Pinus sylvestris*), were also examined in this study for comparison purposes. These wood chips were obtained from International Paper in Kwidzyn, Poland.

Table 1. Soil Properties at the Beginning of the Experiment (March 2008)

Soil parameter	Value	Units
<i>Physical properties:</i>		
sand (> 0.02)	82.3	%
silt (0.02–0.002)	17.7	
clay (< 0.002 mm)	0.0	
<i>Chemical properties:</i>		
pH	6.7	mg per 1 dm ³ of soil
N-NH ₄	8.7	
P	99	
K	91.7	
Ca	460.0	
Mg	75.7	
Cl	12.7	
Soil Organic Carbon	0.40	

Preparation of Fibrous Raw Materials for Chemical Analyses and Pulping

The samples of straws of tall wheatgrass, tall fescue, tall oatgrass, and *Miscanthus* stems and leaves selected for testing were about 5 kg each. The first three raw materials contained plant stems, leave sheaths, and leaf blades. Chemical analyses were conducted on milled fractions of the fibrous raw materials having a particle size of 0.5 to 1 mm. These fractions were obtained by milling of the fibrous raw materials in a Kerner mill and then sieving the resulting flour through a set of sieves with selected mesh sizes. The samples of straws material for milling were collected from various locations of the 5 kg raw material batch. About 100 g of sawdust of each raw material was prepared in the milling process. Two grams samples of sawdust of each raw material were used for the determination of the content of cellulose, lignin, extractives, and ash analysis. These samples were collected from various locations of the samples of sawdust prepared for analyses.

Straw biomasses were prepared for pulping by cutting (manual or mechanical) stems into segments with lengths between 10 to 20 mm. Wood chips were prepared for pulping by screening the chips through a laboratory Santasalo-Sohlberg AB chip sorter (Finland). Wood chips that passed through a sieve with openings 23 mm wide but retained on a sieve with openings 18 mm wide were used.

Removal of Inorganic Components from Sawdust of Fibrous Raw Materials

A weighed portion of sawdust (2 g of oven-dried material) with a particle size of 0.5 to 1 mm was placed in an Erlenmeyer flask. Then, 100 mL of distilled water was poured into the flask and the flask was placed in a water bath and heated for 60 minutes at 50 °C. After this time, the water was separated from sawdust by filtration in a porous P100 funnel and the sawdust was washed with 100 mL of distilled water and dried in a laboratory drier at 105 ± 2 °C to a constant weight. The ash content was determined in accordance with the standard discussed in the next section.

Determination of Properties of Fibrous Raw Materials

The calorific value (as the higher heating value) of the dried biomass straws was measured at Power Research & Testing Company Energopomiar in Gliwice, Poland on behalf of the Institute of Plant Breeding and Acclimatization of Radzików, Poland. The

contents of cellulose, lignin, extractives, and ash were determined in accordance with the PN-92/P-50092 standard (1992). According to this standard for the outcome of the final determination of the content of cellulose, lignin, extractives, and ash, results are given as the arithmetic average of two parallel determinations, the results of which do not differ by more than 0.2%. The results reported in this study are the arithmetic average of the two such replicates.

Determination of Cost of Establishment of One Hectare of Plantation

Costs of establishment of grass grown for biomass were calculated based on an accounting method. All inputs (including labor, oil, chemicals, seed, or plantlets *etc.*) were considered. Field preparation, biomass harvesting, and other necessary operations were all included.

RESULTS AND DISCUSSION

The key criteria for evaluating the suitability of plants as a raw material for combustion are the amount of biomass from one hectare of cultivation, the amount of heat obtainable per unit weight of biomass, the cost of establishment of plantation, and the content of mineral substances determined as ash. The results of the calorific determinations of these indices for biomasses studied are shown in Table 2 and in Fig. 1.

Table 2. Biomass Yield from Grass Crops, their Calorific Value, and the Cost of Cultivation

Plant species	Biomass yield	Heating Value (mean \pm std. deviation) **	Cost of establishment of one hectare of plantation
	(Tons ha ⁻¹) *	(MJ·kg ⁻¹)	Euros (€)
Tall wheatgrass (TW)	6.6–10.4	17.89 (\pm 0.21)	527–600
Tall fescue (TF)	8.4–14.1	17.47 (\pm 0.26)	
Tall oatgrass (TO)	7.5–12.4	18.29 (\pm 0.29)	
<i>Miscanthus</i> (whole plant)	12.2–21.6	18.56 (\pm 0.24)	3957

* Three-year average, differences according to fertilization applied

** Analysis performed by Energopomiar, Gliwice, Poland

The data in Table 2 show that the yields of dry biomass per hectare of cultivation of tall wheatgrass, tall fescue, and tall oatgrass were on average 8.5, 11.3, and 10 t/ha/year, respectively. The yields of these grass straws were noticeably lower than the yield of *Miscanthus* biomass (on average 16.9 t/ha/year). Porensky *et al.* (2014) reported that tall wheatgrass and tall fescue yielded 7.3 to 9.9 and 3.5 to 6.1 tons of biomass per hectare per year, respectively. The yield of tall wheatgrass grown in Poland was thus similar to that grown in the US, while the yield of tall fescue grown in this study was much higher.

The biomass productivity of *Miscanthus* was previously reported by Borkowska and Molas (2013) for Poland and by Lewandowski *et al.* (2000) for northern and central Europe. According to the former authors, the average biomass yield in a 4-year crop cycle was the same as in this study (*i.e.* 16.9 t/ha/year). According to the latter authors,

the yields of *Miscanthus* biomass in Europe may be in the range of 4 to 30 t/ha/year, but higher in the southern regions (e.g., Greece or Turkey at 26 to 44 t/ha/year) than in northern regions. Despite the higher biomass yield per hectare of *Miscanthus* when compared to other grasses examined in this study, it has the highest establishment cost for plantation, growing, and harvesting (Table 2). However, further growing and harvesting costs of perennial grasses grown for biomass are more or less similar in case of species studied. *Miscanthus* × *giganteus* can be grown longer (up to even 20 years after planting) than tall wheatgrass, tall fescue, and tall oatgrass, where the process of plantation actually should be repeated every 3 to 4 years because of the decrease in the yield of biomass to 3 to 4 tons/ha/year after this period of time.

The biomass yields of the grasses studied are several times higher than that of wood. Bodył (2009) and Bowyer (2001) reported that the annual growth of birch and white spruce wood is 3.0 and 1.8 tons per hectare of forest per year, respectively. *Miscanthus*, and especially tall wheatgrass, tall fescue, and tall oatgrass can also compete with fast-growing poplar plantations (varieties Dorscamp, Barn, Beaupre, Hybrid 275), which have yields of 14 to 25 m³ of wood/ha/year in Polish conditions. This biomass yield of poplar is equivalent to 5.3 to 11.5 t/ha/year of oven-dried wood (Bodył 2010).

Table 2 also presents the calorific values of the various grass straws. These data illustrate that tall wheatgrass and tall fescue had calorific values of 17.5 to 17.9 MJ·kg⁻¹, which are comparable to eucalypt wood (*Eucalyptus globulus*) (Telmo and Lousada 2011). Tall oatgrass and *Miscanthus* had higher calorific values (18.3 to 18.6 MJ·kg⁻¹). These calorific values, however, are lower than the values from European wood species. Hardwoods such as beech (*Fagus sylvatica*), oak (*Quercus robur*), and aspen (*Populus euramericana*) have calorific values ranging from 18.7 to 19.2 MJ·kg⁻¹, and softwoods such as seaside pine (*Pinus pinaster*), Douglas fir (*Pseudotsuga menziesii*), and spruce (*Picea abies*) have calorific values ranging from 19.7 to 20.2 MJ·kg⁻¹ (Demirbas 1997; Telmo and Lousada 2011).

Figure 1 shows the ash content of the grass straws with and without washing pre-treatment for 1 h.

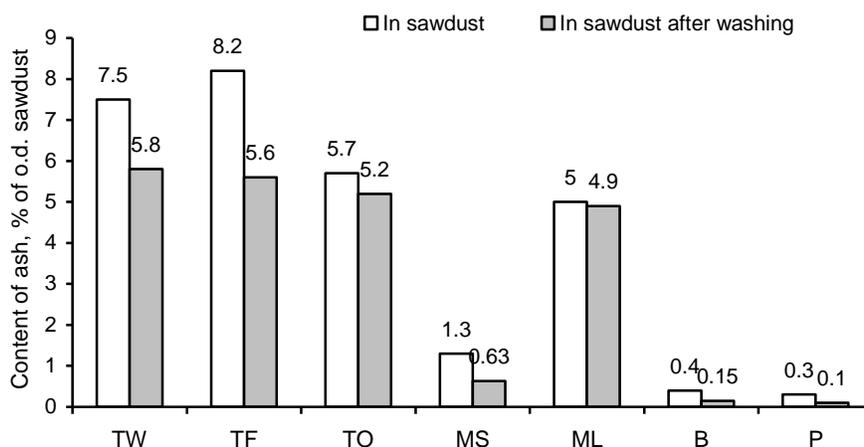


Fig. 1. Ash content of various ground biomass studied before and after washing with water pre-treatment for 1 h

The sources of the inorganic ash are from the uptake of minerals from the soil by the plants, as well as from soil contamination that occurs during harvesting, storing, and

transporting the biomasses. High ash content is a disadvantage for biomass utilisation. This constituent significantly reduces the calorific value of the biomass; it also causes slag formation during combustion. High ash also increases the amount of undesired by-products produced during biochemical processing and contributes to the formation of low-melting point smelt deposits on superheaters in the kraft chemical recovery furnace and inorganic scale deposits on black liquor evaporator surfaces (Turs *et al.* 1992; Olanders and Steeari 1995). In addition, mineral ash created during biomass combustion does not possess much fertiliser value because of its lack of nitrogen content.

Figure 1 shows that the ash content of tall wheatgrass and tall fescue straws was high and constituted 7.5 to 8.2 wt.% (oven-dried mass). Although lower but still high, the ash levels in tall oatgrass and leaves of *Miscanthus* were 5.7 and 5.0 wt.%, respectively. *Miscanthus* stems were characterised by the lowest ash of 1.3 wt.%; however, this level was four times higher than in birch and pine (0.3 wt.%). The ash levels determined in this study for *Miscanthus* grown in Poland are consistent with other published reports (Cappelletto *et al.* 2000; Ververis *et al.* 2004; Brosse *et al.* 2012), while the values for tall fescue were 3 wt.% higher than that reported by Pahkala *et al.* (1997).

This investigation also showed a four-fold higher ash content in *Miscanthus* leaves than in its stems. Pahkala and Pihala (2000) observed a similar trend with tall fescue, as well as reed canary grass, meadow fescue, and goat's rue. Literature sources also showed that one of the ways of reducing the mineral content of biomasses is by crushing and washing it (Turs *et al.* 1992). For this reason, we evaluated the susceptibility of these inorganics to removal by hot water pre-treatment of the ground biomass (Fig. 1).

The data presented in Fig. 1 showed that this pre-treatment could remove 22.7%, 31.7%, 8.8%, 51.5%, 2.0%, 62.5%, and 66.7% of the inorganic compounds from tall wheatgrass, tall fescue, tall oatgrass, the stems and leaves of *Miscanthus*, birch and pine, respectively. The ash in tall wheatgrass and tall fescue was much more susceptible to leaching than the ash in tall oatgrass and *Miscanthus* leaves. However, as can be seen from the data (Fig. 1), the inorganic content in these biomass materials after washing pretreatment was still many times higher than that found in wood (4.9 to 5.8% versus 0.10 to 0.15%).

As mentioned in the Introduction, an alternative use of grass straw is to process the material into cellulose pulps, which could then be used in papermaking. An important factor affecting the suitability of a particular biomass as a papermaking raw material is their lignin and extractives contents. The lignin content significantly affects the fibre isolation process (Petit-Conil *et al.* 1997) and pulp yield (it is generally accepted that birch gives significantly more cellulosic pulp per ton of wood than pine and that this results in part from lower the content of lignin the former raw material). Extractives content also affects pulp yield and contribute to pitch deposits during the making of paper on the paper machine (Allen 1988; del Río *et al.* 1998). The results of analyses of lignin and extractives for grass straws and wood are shown in Fig. 2.

Figure 2 shows that *Miscanthus* stems contained the highest amount of lignin of the grass straws studied (20.6 wt.%). This raw material had more than 2% units more lignin than tall wheatgrass and leaves of *Miscanthus* (18.3 and 18.0 wt.%, respectively), and about 5% units more than tall fescue and tall oatgrass (15.9 and 15.4 wt.%, respectively). The lignin content of *Miscanthus* stems is similar to that of birch wood. In the case of tall wheatgrass and *Miscanthus* leaves, the lignin content is comparable with hemp stems and still lower than in birch wood. Tall fescue and tall oatgrass have lower

lignin levels than either birch wood or hemp stems (Danielewicz and Surma-Ślusarska 2010, 2011). In terms of the lignin content, the tested biomasses appear to be favourable as a fibre source for papermaking. On the other hand, this characteristic may be one of the reasons for their lower heating values than wood. Demibraş (2001) showed, in fact, a highly significant linear correlation between the heating value of the biomass fuel and the lignin content.

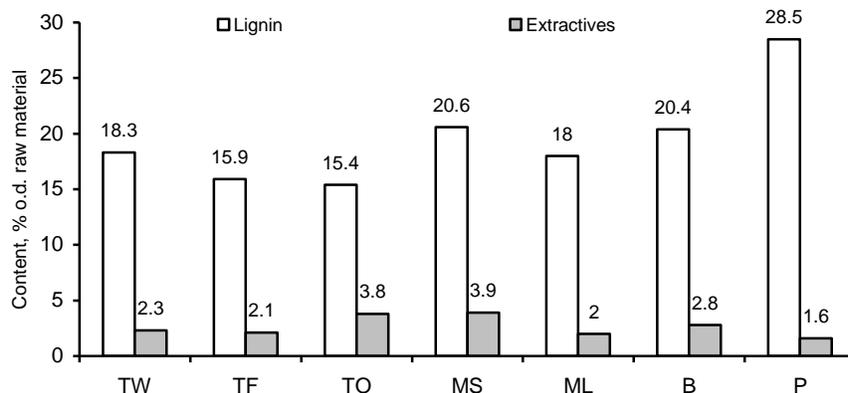


Fig. 2. Lignin and extractives levels in grass straws and wood

The lignin levels in *Miscanthus × giganteus* were studied by Cappelletto *et al.* (2000), Veveris *et al.* (2004), and Brosse *et al.* (2012). The results reported by these authors are, however, quite different. The lignin content in *Miscanthus* examined by the two first authors amounted to 27.4% and 27.0%, respectively, while in the latter study it was 12.3 wt.%. Tall wheatgrass and tall fescue had lignin contents that were 3.7% and 0.7% units lower, respectively, in our study than those reported by Monono *et al.* (2013) and Pahkala *et al.* (1997). No literature reports were found in which the lignin content of tall oatgrass was measured. Biomass materials can be a source of many chemical compounds that are difficult to obtain by chemical synthesis. They can be obtained first of all as turpentine and rosin soaps in pulp mills in such operations like degassing of digester and scrapping of rosin soaps from the surface of black liquor. Such compounds can also be isolated from the biomass as essential oils by subjecting them to grinding and steam distillation or organic solvent extraction (Covey *et al.* 2014).

Figure 2 shows that the grass biomasses contained comparable amounts of extractives to seasoned birch and pine chips. Tall oatgrass straw and stems of *Miscanthus* contained more extractives than the other straw grasses (3.8 to 3.9% versus 2.1 to 2.3%).

Another constituent of that determines the usefulness of a fibrous biomass for pulp production is its cellulose content. In this study, the cellulose content was measured using the Seifert method, wherein the cellulose is separated from the plant material in the process of its processing with aqueous solutions of acetylacetone, dioxane, and hydrochloric acid (Fig. 3) (Seifert 1960). In addition, taking into consideration the high mineral contents in straws of grasses, these components in the “Seifert” cellulose were also determined (Fig. 3). It is shown that the ash content in the uncorrected “Seifert” cellulose extracted from tall wheatgrass, tall fescue, tall oatgrass, and leaves of *Miscanthus* ranged from 8.3 to 17.4 wt.%. Therefore, the uncorrected cellulose content in the grass straws were corrected by subtracting the ash contents from this value (the first column in Fig. 3), which yielded a corrected (real) cellulose content.

Straws of tall wheatgrass, tall fescue, tall oatgrass, and stems and leaves of *Miscanthus* contained pure cellulose contents of 35.3%, 28.6%, 29.3%, 38.9%, and 34%, respectively. Thus, *Miscanthus* stalks contained the most of cellulose, followed by tall wheatgrass and leaves of *Miscanthus*, whereas tall fescue and tall oatgrass contained the least of this chemical component.

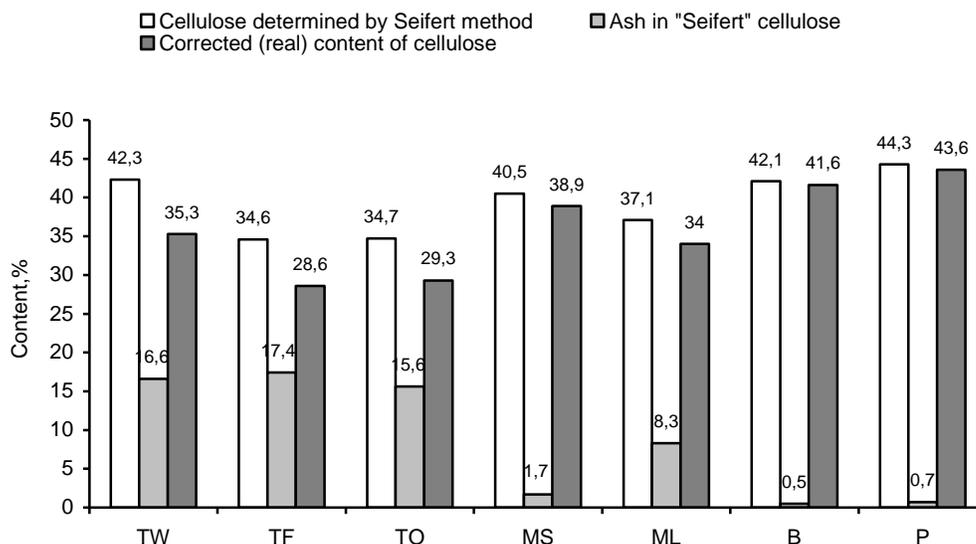


Fig. 3. Cellulose content in various biomass straws by the Seifert method (uncorrected), the ash content in "Seifert" cellulose, and the corrected (real) cellulose content

The content of cellulose in the giant *Miscanthus* was also studied by Cappelletto *et al.* (2000), Ververis *et al.* (2004), and Brosse *et al.* (2012), while Pahkala *et al.* (1997) and Godin *et al.* (2010) determined the cellulose content in tall fescue. The present results for this component in *Miscanthus* stalks were similar to the results obtained by the first two authors; however, they differ from those reported by Brosse *et al.* (2012), who state that the cellulose content in this plant material is 51.2%.

The present results for determination of cellulose in tall fescue were also lower by approximately 5% than the results reported by Pahkala *et al.* (1997) and Godin *et al.* (2010). This is probably due to the fact that these authors did not recognise the high mineral substances content in tall fescue and that these substances tend to remain attached to the solid (cellulose) fraction when the separation method is based on the dissolution of the rest of the organic material, as reported by Anglés *et al.* (1997).

CONCLUSIONS

1. The biomass yields of dry straw per hectare of cultivation of tall wheatgrass, tall fescue, and tall oatgrass were lower than *Miscanthus* by 47.9, 33.1, and 40.8 relative %, respectively. However, the grasses have a much lower cost of establishment for plantation when compared with *Miscanthus*.
2. Tall wheatgrass, tall fescue, tall oatgrass, and leaves of *Miscanthus* contained 5% to 8% inorganic ash, compared to 0.3% to 0.4% for wood and 1.6% for *Miscanthus*

stems. The mineral contents of the ground straws were reduced by 2% to 50% by washing them with water under suitable conditions.

3. Tall wheatgrass, tall fescue, and tall oatgrass have lower lignin content than *Miscanthus* stalks and birch and pine woods.
4. The contents of pure cellulose in the oven-dried grass straws, particularly tall fescue and tall oatgrass, were lower than in wood.

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